# Requirements for Improved Detectors in Macromolecular Crystallography

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## Important players in Synchrotron Detectors

- **Uli Arndt,** Cambridge, UK – Invented the modern Rotation Method, which is how we take data. Pushed video detectors. Conceived first commercial synchrotron detector
- **Jules Hendrix,** Hamburg – Commercialized an automated imaging-plate camera
- **George Reynolds, Sol Gruner,** Princeton – Parallel video developments. Moved to CCD work.
- **Michael Strauss, Ed Westbrook, Steve Naday,** Argonne – Large array CCD-based detectors
- **Walter Phillips, Marty Stanton,** Brandeis – Important mechanical and electronic advances
- **Ron Hamlin, Chris Nielsen** – Commercialized large-array detectors
- The multi-wire Guys: **Perez-Mendez, Xuong, Hamlin, Charpak, Kahn**

## What are the goals of the crystallographic experiment?

Bring all Bragg planes into diffracting position

*Integrate* the diffraction intensity from each Bragg reflection

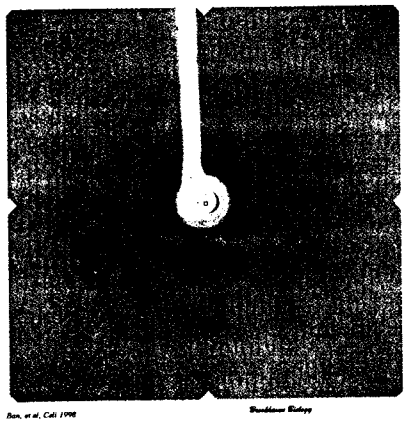
- Through the crystal rocking curve
- Over the reflection area on the detector face

To accomplish this

- Read out detector frequently as the crystal rotates
- Resolve spots on the surface of the x-ray detector

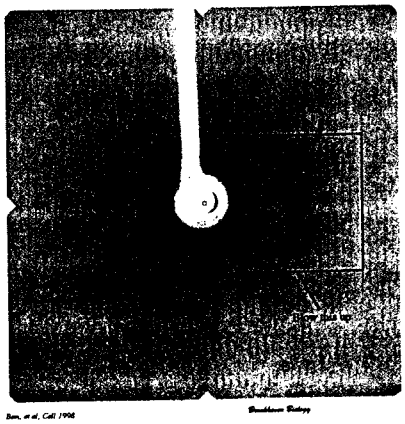
**Here's an example of a difficult problem!**

The longest unit-cell edge is 570Å in this photo from crystals of the 50S ribosomal subunit.



Ban, et al, Cell 1998

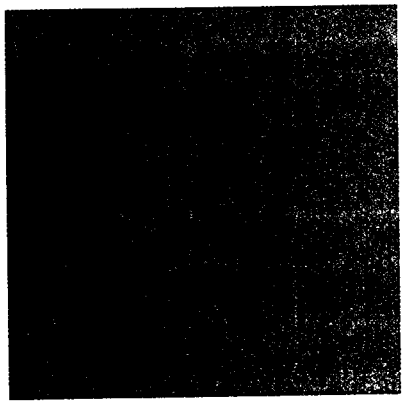
Let's look at it in detail:



Ban, et al, Cell 1998

**The challenge for the Detector Maker:**

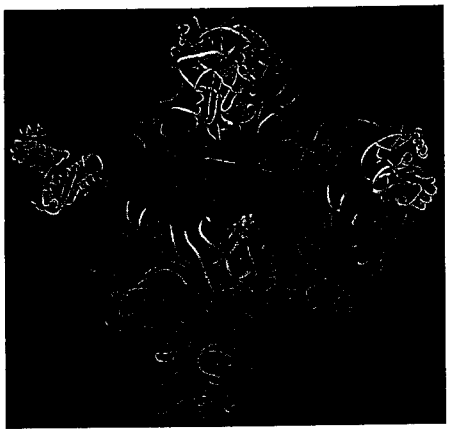
One needs to measure data like these quickly, without adding width or noise to the spot.



Ban, et al, Cell 1998

## The 50S Subunit

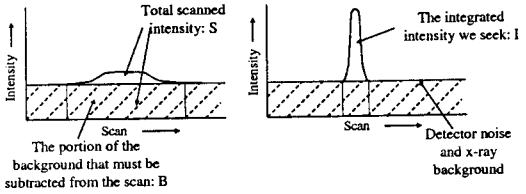
When it works, the payoff is great. This is a simplified cartoon of the 100,000 atoms in the 50S subunit of the ribosome.



## What are the issues in detector specification?

- Individual diffraction spots must be sampled finely, and they must not overlap (small point spread)
- No extraneous noise may be added to the intensity measurement
- Readout must be quick
- The dynamic range of measurement must be large

## Quantitate the advantage of small point-spread and low detector noise




- We have: $I = S - B$
- Standard error propagation gives $\sigma^2_I = \sigma^2_S + \sigma^2_B$
- Since I, B, and S all are counts, $\sigma^2_S = S$, $\sigma^2_B = B$
- This gives: $I / \sigma_I = (S - B) / (S + B)^{1/2}$

## Evaluate Detective Quantum Efficiency (DQE) for an Integrated Intensity

$$\varepsilon = \frac{\left(I/\sigma\right)^2_{out}}{\left(I/\sigma\right)^2_{in}} = \left(\frac{I/(S+B)^{1/2}}{\sqrt{I}}\right)^2 = \frac{I}{S+B} = \frac{I}{I+2B}$$

So any change in an experimental system that decreases B, whether it's by ***keeping detector noise low*** or ***keeping reflections sharp***, improves the efficiency of this system.

## Now let's look at how the detectors we use have evolved

## X-Ray Film as a Detector

Many crystal structures were solved with the Rotation Method of data collection on film.

*Good points:*

- Inexpensive when that seemed important
- Pretty linear
- Excellent spatial resolution

***Bad points:***

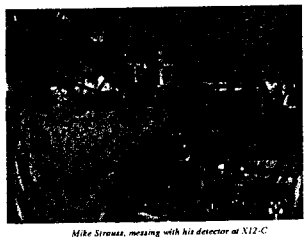
- Poor dynamic range
- ***Extremely*** inconvenient

*Messing with an Arndt/Wonacott Rotation Camera, NSLS Beamline X12-C. Circa 1986.*

## Early Argonne CCD detector

**Shown being tested at NSLS beamline X12-C in 1987.**

Light chain is:
- Phosphor
- 115:40mm fiberoptic taper.
- 2-stage image intensifier
- *f* 1.0 Lens
- Tektronics 512x512 CCD

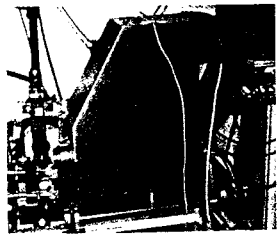


*Mike Strauss, messing with his detector at X12-C*

## Early Brandeis CCD detector

**Shown while being tested at BNL in early 1989.**

Its (folded) light chain is:
- Phosphor converter
- 75mm image intensifier
- 75:38mm fiberoptic taper
- 40mm image intensifier
- 38:18mm fiberoptic taper
- Mirror
- 50mm *f* 2 macro lens
- 1.3k x 1.0k Kodak CCD (7 micron-pixels).



*Again, on the rotation camera at X12-C*

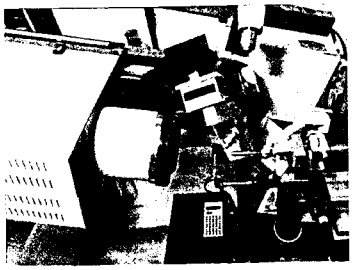
## The Nonius/Arndt FAST detector

**Installed at BNL late 1989**

It has a phosphor on an image intensifier, a fibre-optic taper, and a Silicon-Intensifier-Target video tube.

*Good points:*
- Single-photon counting
- 100% duty cycle
- Nearly the first automatic data-collection system.

*Bad points:*
- Narrow dynamic range
- Few pixels.

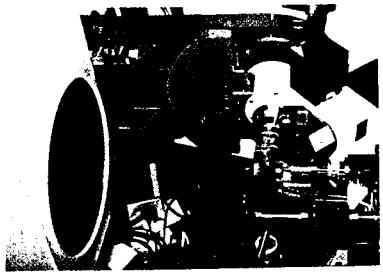


*The FAST in the X12-C hutch*

## The most successful synchrotron detectors have been the MAR imaging-plate systems

*Good points:*
- Fairly efficient
- Quicker than Film
- Many pixels relatively inexpensively
- One of the first automatic data-collection systems

*Bad points:*
- Slow readout
- Not *really* efficient
- Only decent point spread

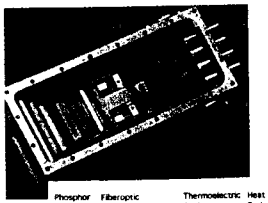


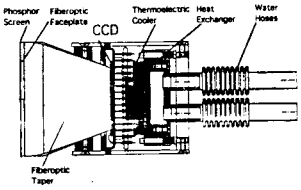
*The MAR300 detector on the FAST diffractometer at X12-C*

## Direct-coupled CCD-based detectors

**An early model is this one from Argonne, used at NSLS X14 in 1992.**

The light chain:
- Phosphor converter
- 43 x 43 mm to 25 x 25mm fibre-optic taper
- 25 mm square 1,024 CCD.

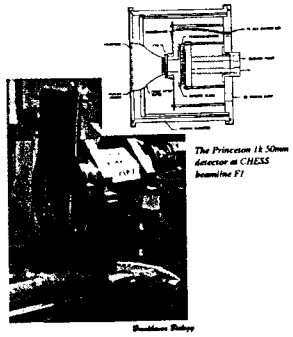





## Direct-coupled CCD-based detector from Princeton

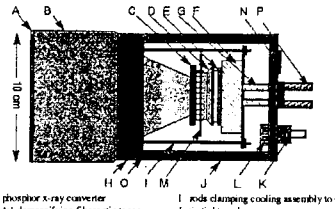
**The first such detector "permanently" installed at a synchrotron beamline, Spring 1993**

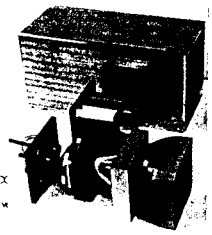
The light chain:
- $Gd_2O_2S$:Tb phosphor
- 50 x50 mm to 20 x 20mm fibre-optic taper (2.6:1 ratio)
- 1024 x1024 pixel CCD chip



*The Princeton 1k 50mm detector at CHESS beamline F1*

## The Brandeis University mechanical design for CCD-Based X-Ray Detectors has become popular



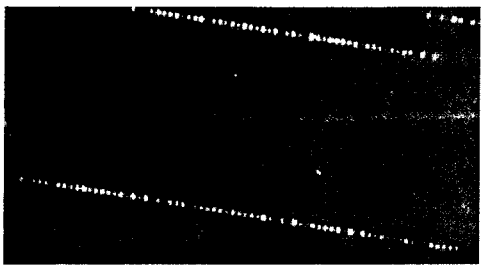
A phosphor x-ray converter
B 4:1 demagnifying fiberoptic taper
C CCD and socket
D heat transfer block
E thermoelectric cooling module
F water-cooled heat exchanger block
G cooling-water tubes
H flange bonded to fiberoptic taper
I rods clamping cooling assembly to OC
J air-tight enclosure
K evacuation / dry-air fill tube and valve
L desiccant container
M preamplifier
N electrical feed-through
O "O" ring seal
P flexible insulated water tubing

*W. Phillips and M. Stanton*

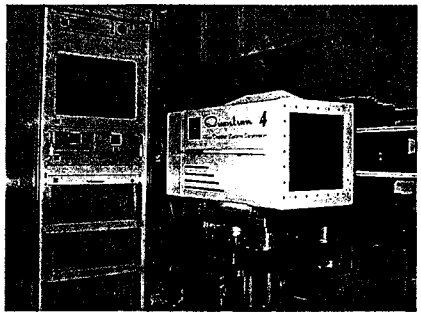
## Two different models of Brandeis detector are in constant use at the NSLS

## The Brandeis 2x2 array (The B4) has been used for many large unit-cell projects

The pattern here reveals a 720Å spacing from the cytochrome bc1 complex. This sort of problem exemplifies the need to resolve many orders across the face of the detector.



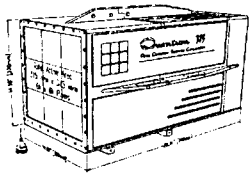
## The most successful commercial vendor of this sort of detector is Area Detector Systems Corp.



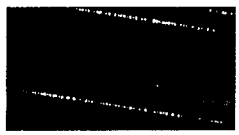

*The four-module Quantum-4 detector, by ADSC, is in use at NSLS X12-B.*

## ADSC are attempting to push the state of this art with their Quantum 315

**Active area:** 315mm square
**Number of pixels:** 6k square
**Pixel size:** 51 x 51 microns
**Spatial resolution FWHM:** 90 microns, possibly 200 microns at 1%
**Taper ratio:** 3.7 to 1
**CCD type:** 2k x 2k
**Readout times (no data collected!):**
(Full Resolution): 1 second
(2x2 binned): 330 ms
**Dark current (@-50C):** 0.002 Xph/pixel/sec
**Read Noise:** (1 MHz pixel rate): ~2 Xph
**Full well:** ~30,000 Xph/px




## If this is the state of the art, what do we need?



Spatial resolution: Typical diffraction-spot sizes from new synchrotrons are on the order of 100-200 microns.

- *Sampling*: We need to sample these 2-3 times to see the profile. 50 microns is good.
- *Spreading*: No spread is good. Wings of spread should fall quickly. Want < 1 Pixel

## Dynamic range, Maximum count rate:

These detectors are "integrating" detectors, so counting "rate" doesn't really mean anything.

For a REALLY big spot, a detector at an APS undultor beamline sees about 100,000Xph/s in the central pixel. This is beyond the well depth of existing detectors. Nonetheless, one might hope for a *maximum count rate* of $10^6$ Xph/px/s

The x-ray background in that pattern may be 20-100 Xph/s. A *dynamic range* in the scale of 30-50,000 is appropriate.

Given a finite well depth, the maximum count rate then depends on the framing rate.

## What about read-out speed?

*Detector dead time:*

- Would like this to be as small as possible.
- With double buffering this could be zero

*Maximum framing rate:*

- Don't need more than ~3000 Xph in a typical peak to integrate well.
- May need to frame every few tenths of a second with a bright beam *or very much faster for special cases.*

## Maximum number of orders:

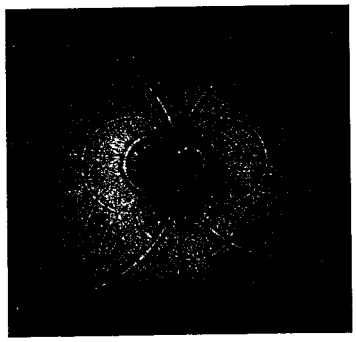
One would like always to work in "normal-beam" mode to use the top and bottom of the detector equally.

Biologists' wildest fantasies might suggest 1000 Å unit cell size at 2 Å resolution:

- 500 orders above and below the beamstop, or 1000 orders total.
- One needs 3-4 pixels to sample a spot profile, 2-3 pixels to measure background.
- Say 6 px/reflection, or 6000 resolution elements.

Remember that the useful width sampled by a group of $n$ pixels is $n$ times the width of each pixel plus the point-spread function of one pixel *at about 1% of maximum.*

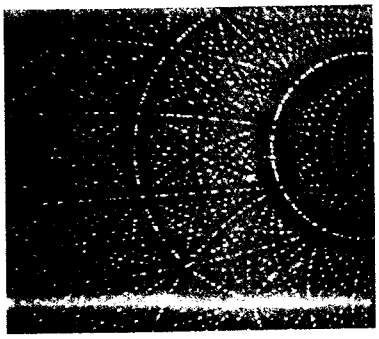
## A challenge for detector builders is Laue photography



This method can be used for kinetic crystallography, to capture states in a reaction pathway.

The Laue patterns are almost fractal; the detail can be very fine.

The intensity is limited by the spectral brightness of the source, but microsecond framing could be useful.



## Requirements for an Improved Detector for Macromolecular Crystallography

| Property | What we'd like | What we have |
|---|---|---|
| Pixel Size | 50 $\mu$m | 50 $\mu$m |
| Point Spread | <1 pixel | 90/200 |
| Dynamic range | 50,000 | 30-50,000 |
| Maximum count rate | $10^6$ Xph/px/s | ~$10^5$ Xph/px/s |
| Detector dead time | near zero | 0.3 sec |
| Shortest framing time | a few tenths of a second | 0.3 sec |
| Number of diffraction orders | 1000 | 500-600 |